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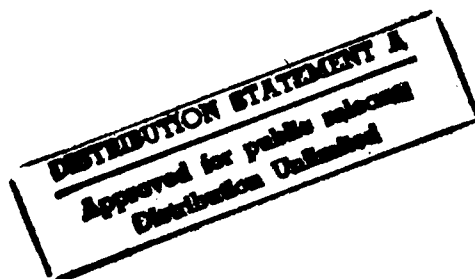
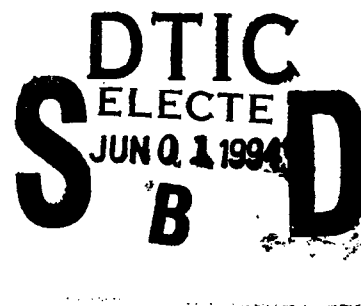
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The In Situ Observation of Epitaxial Diamond Thin Film Nucleation  
and Growth Using Emission Electron Microscopy

Submitted by

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13. ABSTRACT (Maximum 200 words)  A method for activation of high resistivity,(100) texture CVD diamond films with gold, to improve low field electron emission is described. A model based on the dielectric breakdown of the diamond film is proposed and atest experiment, which consists of heating the gold layer to a point where the gold forms sub-micron sphereoids on the diamond surface, is described which supports the model.  The deposition of carbon and sulfur on Mo(310) is characterized with scanning Auger Microscopy. Correlation between Photoelectron emission Microscopy, scanning Auger Microcopy and Auger spectroscopy can be made, so that individual features in PEEM and SAM images can be identified by elemental composition.  The initial design of a Seeded Supersonic Molecular Beam system for diamond deposition is described.					
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## Summary of Progress:

The experimental goals of our project center on (1) the preparation of substrates with high step-terrace density that are suitable templates for diamond heteroepitaxy, (2) development of a two-dimensional diamond growth method (3) observation of the diamond (100)/H reconstructed surface and (4) the delivery of hydrocarbon reactants for diamond deposition at controlled kinetic energy and energy spread at high enough flux to grow diamond and illuminate the process for in situ observation and optimization.

The time period covered in this report was dedicated to new construction of two large additions to our PEEM apparatus, the examination of {100} textured diamond thin films for low field electron emission, and preparation of the Mo(310) surface. The choice of Mo(310) is not directly related to the study of Si(310), since Molybdenum is BCC. We have more extensive experience with growth on molybdenum surfaces.

### 1.1 Activation of CVD Diamond Field Emitters

A copy of a manuscript submitted to Applied Physics Letters entitled " Electron Emission from CVD Diamond and Dielectric Breakdown" is appended.

### 1.2 Preparation of stepped/facetted Mo(310) surfaces

An issue central to the growth of defect-free, electronic grade heteroepitaxial growth of CVD diamond is the role of "high energy" sites on the surface, such as steps, which may be equivalent to the "scratches" that are commonly found to nucleate diamond. It may be possible to nucleate diamond at a step, then grow the CVD heteroepitaxial diamond layer laterally across the surface by the "step flow" mechanism.

We have examined Si(310), which may form a stable facetted surface that contains mono-atomic height (100) and (111) faces that could be used for CVD diamond heteroepitaxy.

Although the Molybdenum lattice is BCC, we have a Mo(310) crystal that we have prepared in order to examine diamond growth on a substrate with which we have more experience.

The two dimensional growth fronts observed on Mo(100) have also been observed on Mo(310) (page 7), with the possibly significant discovery that sulfur is present (see page 8, SAM, page 9, SAM + AES). The sulfur is detected in Scanning Auger Spectroscopy, but not with ESCA/XPS. The presence of S in CVD diamond grown on Mo surfaces is probably a widespread occurrence, but relatively unknown due to the apparent low sensitivity of ESCA to sulphur. Another laboratory (CWRU) has reported the presence of S on CVD diamond, also detected by AES, and not in ESCA.

## 2.0 New construction:

### 2.1 Scanning Auger Spectroscopy

A Scanning Auger (SAM) attachment was purchased from combined ONR (1/3) and Ohio University Matching funds (1/3) and a competitive internal equipment grant program "1804 Fund" (1/3) at a total cost of \$32,000. Some of the construction details are shown on pages 10 - 12.

### 2.2 Seeded Molecular Beam System

A schematic of the Seeded Molecular Beam System is shown on page 13. The components are a combination of rebuilt Varian diffusion pumps, rebuilt mechanical backing pumps and standard ISO components. The vacuum system is complete, the nozzle/skimmer assembly will be constructed in 1994.

### 3.0 Publications/Presentations

The following papers have been accepted by Diamond and Related Materials, but have not yet been published.

1. "An In Situ Observation of CVD Diamond Etching and Dissolution Using Photoelectron Emission Microscopy," C. Wang, J. Macaulay, J.D. Shovlin and M.E. Kordesch, Diamond and Related Materials, 1994.
2. "Removal of Non-Diamond Carbon from CVD Diamond Surfaces", W. Engel, D.C. Ingram, J. Keay and M.E. Kordesch, Diamond And Related Materials, Letter to the Editor, 1994.

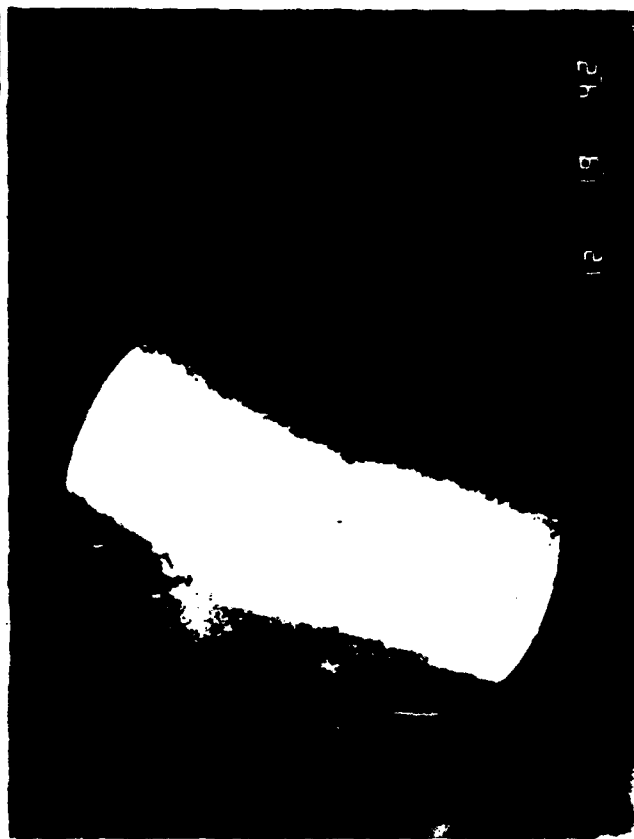
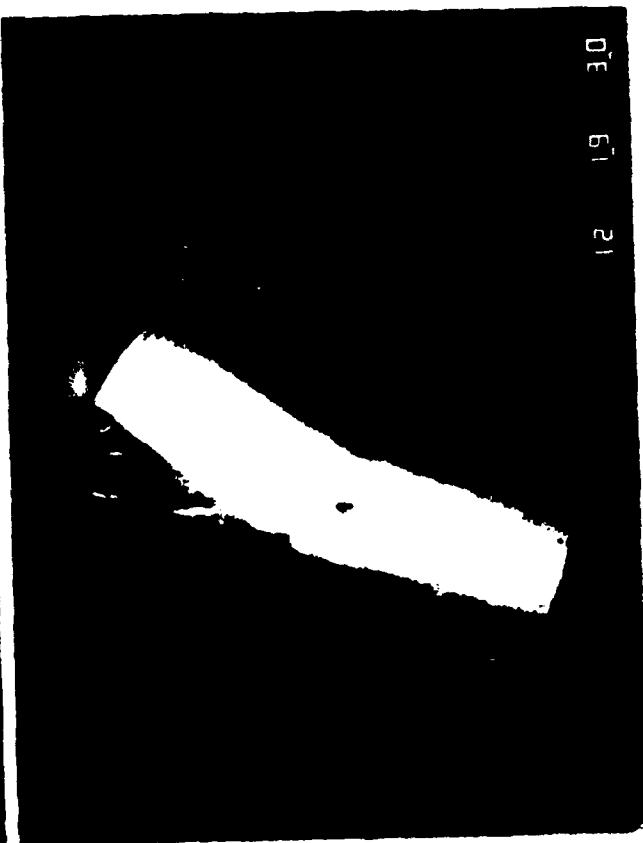
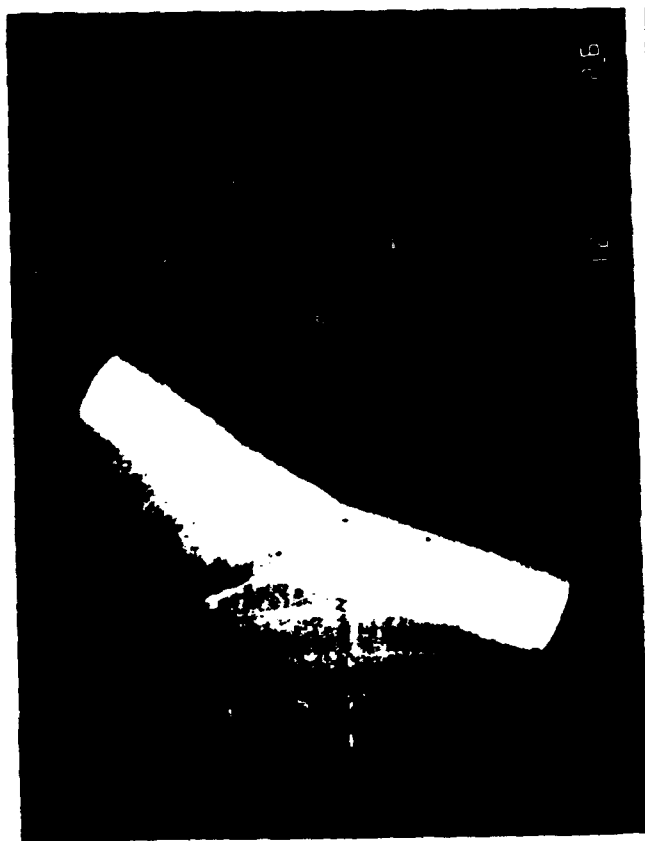
#### Presentations:

1. Introduction to Emission Electron Microscopy for the in situ Study of Surfaces, Martin E. Kordesch, Invited Tutorial Lecture, 51st Annual Meeting of the Microscopy Society of America, Cincinnati, OH, August 1993.
2. Diamond Field Emission Cathodes, J.C. Twichell, M.W. Geis, C.O. Bozler, D.D. Rathman, N.N. Efremow, K.E. Krohn, M.A. Hollis, R. Uttaro, T. Lyszczarz, M.E. Kordesch and K. Okano, Diamond Films 93, Albufiera, Portugal, 1993, unpublished.

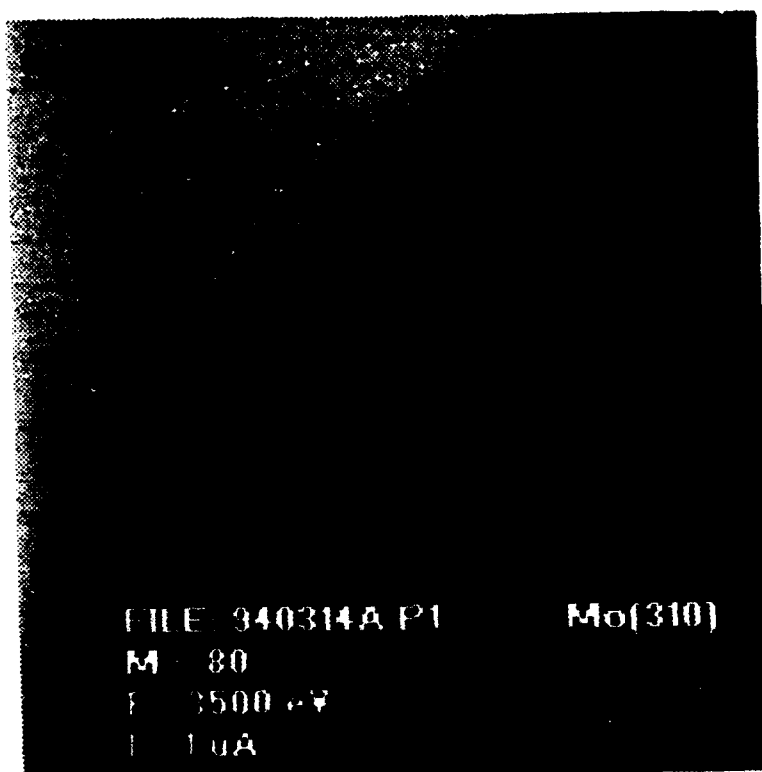
#### Abstracts:

1. "Emission Microscopy of Low Field Cold Electron Emission from CVD Diamond Films", J.D. Shovlin and M.E. Kordesch, APS March Meeting, Pittsburgh.
2. "In Situ Photoelectron Emission Microscopy of Oxidation and Reduction of Mo(100) and Mo(310) at 1000K", A. Garcia and M.E. Kordesch, APS March Meeting, Pittsburgh.

# PEEM micrographs of reaction fronts on Mo(310)

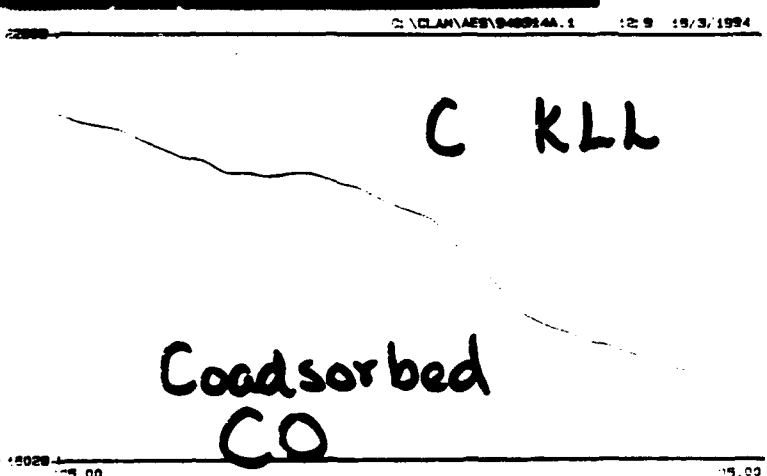
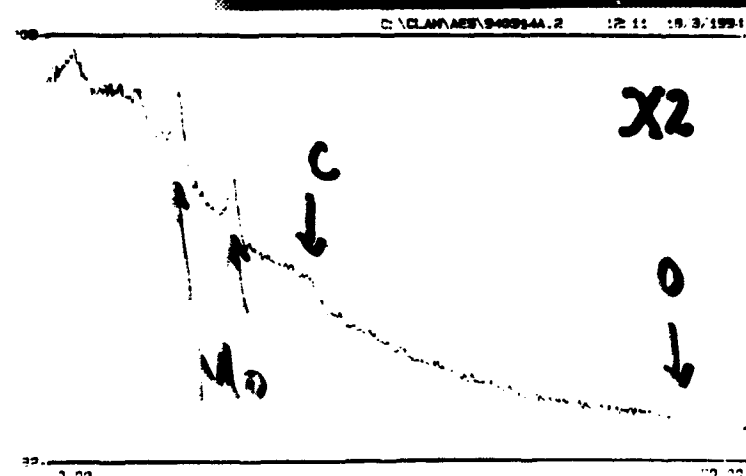
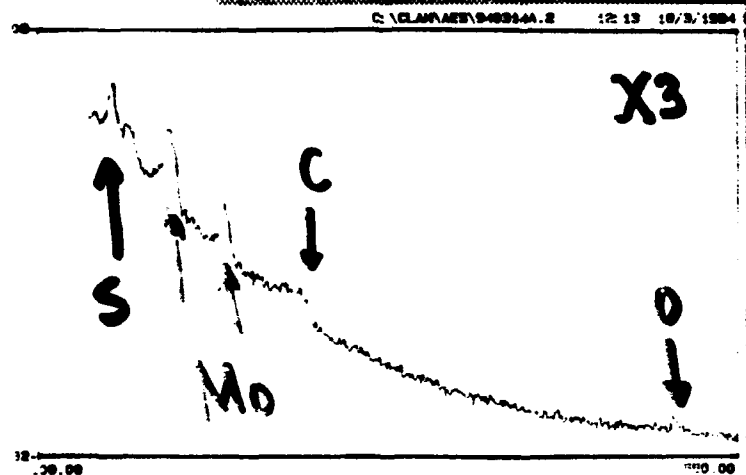
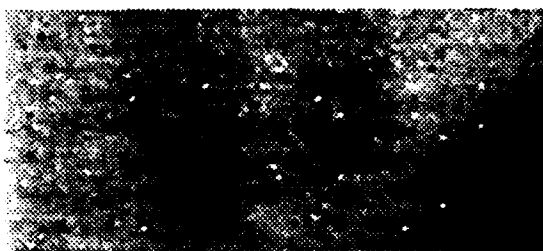
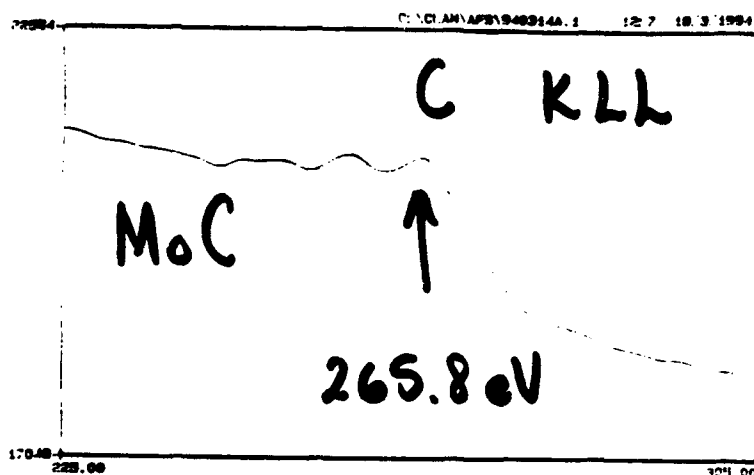
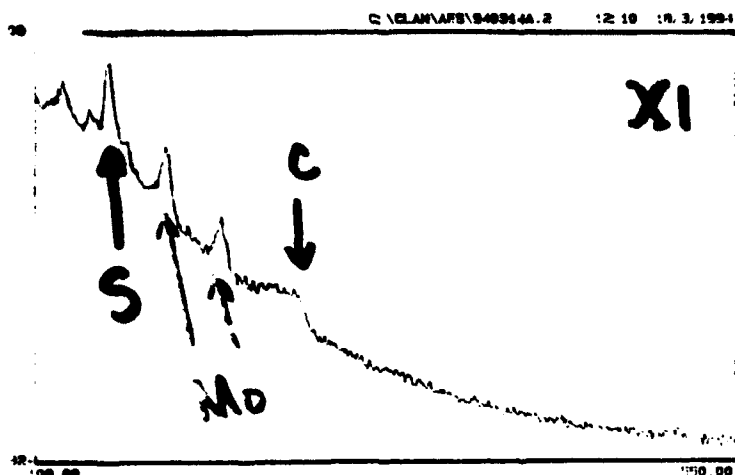


**Scanning Auger micrograph of a front  
boundary at room temperature**



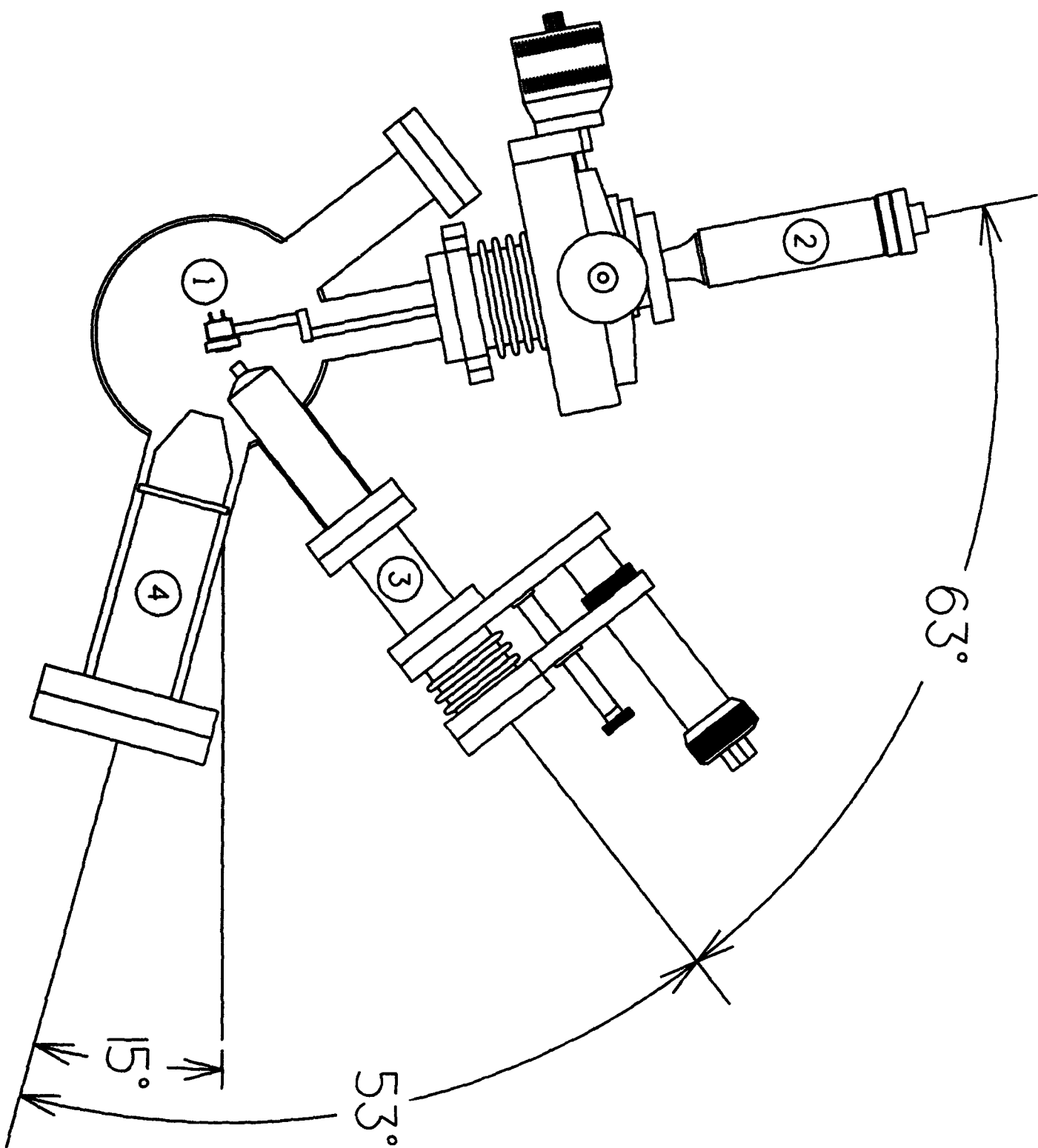
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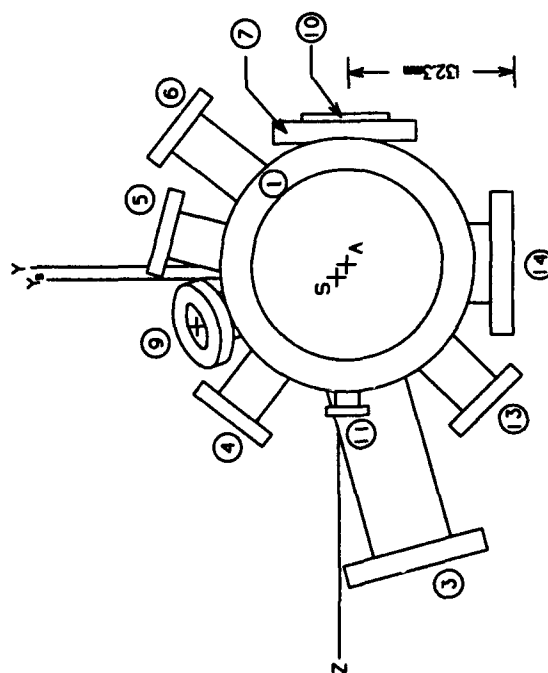
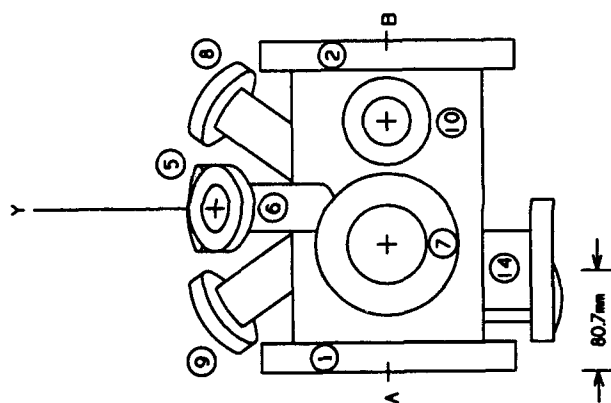
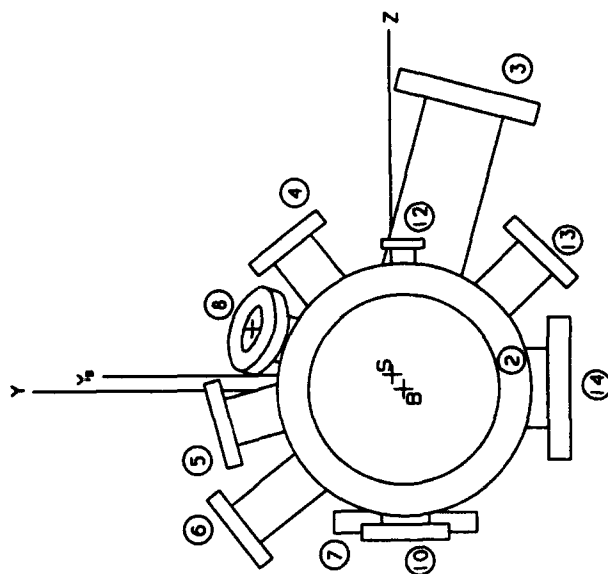
# Results: SAM - AES correlation

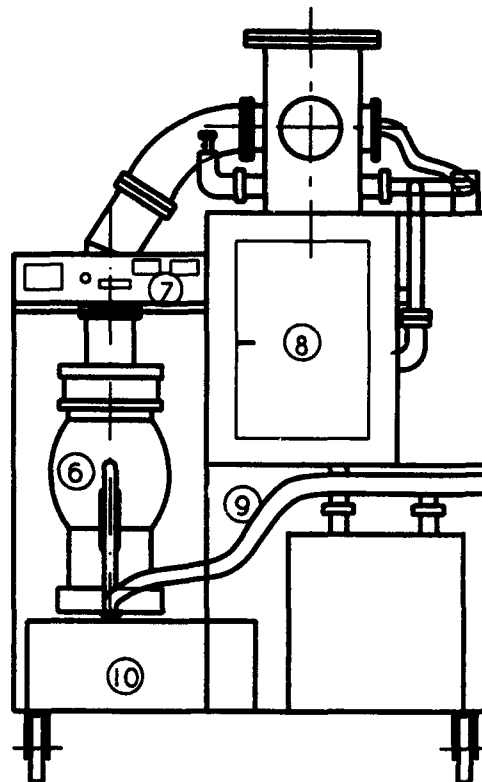
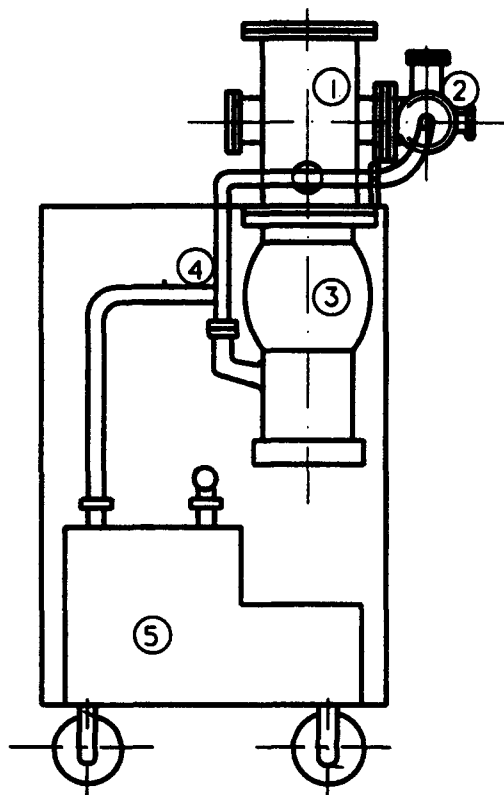


A schematic diagram of a microscope with numbered parts 1 through 9. 1: Eyepiece, 2: Objective lens, 3: Ocular tube, 4: Eyepiece holder, 5: Light source, 6: Stage, 7: Base, 8: Light switch, 9: Stage clips.

- 10







- 1-Expansion chamber
- 2-Skimmer chamber
- 3-Diffusion pump VHS-6
- 4-Fore line
- 5-Mechanical pump for VHS-6
- 6-Diffusion pump VHS-4
- 7-Control panel
- 8-Electrical wiring box
- 9-Exhaust line
- 10-Mechanical pump for VHS-4

# MOLECULAR BEAM APPARATUS

Abstract submitted  
for 1994 March Meeting  
21-25 March 1994

Suggested Session Title:  
Diamond Growth, Properties and Applications

Sorting Category:  
15f

**Emission Microscopy of Low-Field Cold Electron Emission from CVD Diamond Films.** J.D. SHOVLIN, M.E. KORDESCH, Ohio University.<sup>†</sup> — Polycrystalline, (100) textured, free standing, Si and Mo backed, Hot filament-, Flame- and Microwave plasma-deposited, and doped films have been examined in an emission microscope with an applied field of 30–50 kV/cm. Images of a wide variety of CVD diamond substrates, graphite and natural diamond have been obtained that show the location and relative intensity of electron emission in the field emission, photoemission and secondary electron illumination modes. The so-called negative electron affinity (NEA) of the boron-doped IIb diamond (111) surface<sup>1</sup> is often used to account for the low-field cold emission from CVD diamond. The NEA mechanism for electron emission is examined in light of the variety of specimens for which this emission is observed. We present a method of diamond film activation that indicates that dielectric breakdown of the diamond film is responsible for the “low-field” electron emission.

<sup>†</sup> Supported by ONR through BMDIO/IST No. 00014-91-J-1596.

<sup>1</sup> F. J. Himpsel *et al.* Phys. Rev. B 20, 624 (1979).

Prefer Standard Session

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Abstract Submitted  
for the 1994 March Meeting  
21-25 March 1994

Suggested Session Title:  
Photoelectron Emission Microscopy  
and Low Energy Electron Microscopy

March Sorting  
Category:  
29e

In Situ Photoelectron Emission Microscopy of Oxidation and Reduction of Mo(100) and Mo(310) at 1000K. A. GARCIA and M.E. KORDESCH, Ohio University. - Photoelectron emission microscopy (PEEM) has been used in conjunction with scanning Auger microscopy (SAM) to characterize the oxidation of Mo(100) and Mo(310) in situ and in real time with post-reaction spectroscopic imaging using SAM. Homogeneous oxidation is observed in PEEM, however, AES shows that the reduction of the oxide takes place through intermediates identified by the corresponding O KLL peaks at 508 and 510 eV. PEEM shows that that the reduction reaction proceeds as a two dimensional front.  $H_2/CH_4$  mixtures also reduce the oxide layer with subsequent C deposition. The significance for atomic layer epitaxy of diamond is discussed.

Prefer Standard Session

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## Electron Emission From CVD Diamond and Dielectric Breakdown

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### Abstract

After coating with a smooth, non-reactive, sputtered gold layer, previously non-emitting CVD diamond films are observed to emit electrons in an applied field of 30 - 50 kV/cm in an electron emission microscope. The gold overlayer increases the effective field strength applied to the film. The consequent dielectric breakdown of the film provides conductive channels for the observed electron emission.

## Introduction

Chemical Vapor Deposited diamond (CVD diamond) is presently used primarily for heat spreaders and abrasives. The observation of a negative electron affinity for the semiconducting type IIB C(111) surface [1] and a strong interest in diamond as a wide band-gap semiconductor has led to the investigation of the suitability of CVD diamond for a variety of electronic devices [2-4]. Among these applications are robust cold cathodes and flat panel video display devices, which could benefit from the (hoped for) less stringent vacuum requirements for CVD diamond emitters relative to metal [5] and alkali metal-activated emitter arrays [6], and low power consumption relative to thermionic devices.

Central to the optimization of CVD diamond emitters for these applications is an understanding of the emission mechanism, i.e. the origin of both the (apparent) low barrier to electron emission and the conduction pathways through what should be considered an excellent insulator.

Low field, room temperature electron emission from individual polycrystalline CVD diamonds and films on molybdenum and silicon substrates has been demonstrated and observed in emission microscopy by Kordesch and co-workers [7-9]. Typical applied electric fields for such emission are 3-5 MV/m, and current densities range from 1-10 mA/cm<sup>2</sup>. The low field, cold emission (LFCE) from CVD diamond has been observed by other means [10,11], confirming the original observation reported in ref. [8]. Much higher electric fields have been applied to CVD diamond [12] and "amorphous" diamond [13].

In this letter we report a method for inducing LFCE in a partially oriented ( [100] texture), unbacked CVD diamond thin film that did not exhibit LFCE in the as-received state. The CVD diamond film is, however, easily observed under UV illumination, where it is photoconductive. The activation method increases the applied electric field at the surface of the CVD diamond film by

decreasing the distance over which the field is applied. A metal layer is deposited on the CVD diamond film; it is expected that this layer should quench any purely surface effects such as negative electron affinity, suggesting that LFCE in CVD diamond is due to dielectric breakdown of the diamond films.

## Experimental

The emission microscope and vacuum system is a Bauer-Telieps style Low Energy Electron Microscope (LEEM), and is described in ref. 16 and 17. In this experiment the microscope was run in Photoemission Electron Microscopy (PEEM) mode. When observing field emission the ultra-violet lamp was turned off.

An electric potential of 0-19 kV could be applied between the sample and the first lens element, at a distance of approximately 4 mm. A tungsten filament behind the sample was used to outgas the sample and holder through radiative heating, or, upon application of high voltage between the filament and sample, heat the sample through electron bombardment. The sample holder could be cooled with a liquid nitrogen filled reservoir.

Samples analyzed were a free-standing textured [100] Microwave Plasma CVD (MWPCVD) diamond film (Astex, Applied Science and Technology, Inc.), and a previously examined LFCE consisting of a predominately (111) faceted MWPCVD silicon backed film grown in-house. Both samples were pre-treated with a chromic acid/sulfuric acid wash to remove non-diamond surface carbon. The gold coating was deposited with a PELCO Sputter Coater at 300 K; the film thickness is an estimate based on the sputter rate. No independent measurement of the layer thickness was made.

## Results

The textured [100] diamond sample was observed in the PEEM and field emission modes both before and after outgassing to remove adsorbed water. In fields of up to 50kV/cm the film did

not exhibit field emission. The film was removed and coated with 15 nm gold. Upon reinsertion into the vacuum system, the sample exhibited field emission in applied fields above 30kV/cm. Electron emission was observed both before and after sample outgassing for 100 hrs to eliminate the possibility of any trapped gases from the sputtering process interfering with the field emission image. The microscope residual gas pressure during observation was about  $1 \times 10^{-8}$  Torr. A typical field emission image is shown in figure 1. The experiment was repeated for a second [100] sample, where it was found that a thinner layer (5-7 nm) was not sufficient to induce emission. Re-coating with an additional 7 nm of gold produced LFCE.

As a further test of the emission phenomenon, the sample holder was cooled by a liquid nitrogen coldfinger. The field emission image was not changed by cooling.

The sample was heated by electron bombardment to c. 1000 K and then allowed to cool to 300 K. Although the gold remained on the sample, field emission was no longer observed.

Ex-situ Scanning Electron Microscope (SEM) analysis was performed on the gold coated diamond. Figure 2 shows SEM micrographs of the gold coated diamond surface (2a) before and (2b) after heating. Figure 2c is a close-up of a diamond [100] face after heating. From the SEM micrographs it can be seen that prior to heating the gold coating was smooth and uniform, and after heating was broken up into discontinuous sub-micron sized spheroids.

A predominately (111) faceted sample was also observed in PEEM and field emission modes. This sample exhibited field emission in applied fields over 30kV/cm prior to and after gold coating. Heating caused the gold to ball up on the surface (as observed in ex-situ SEM micrographs) but the field emission image remained unchanged.

A graphite disk mounted identically to the diamond specimens was not observed to emit electrons in the "clean" or gold coated state, both before and after gentle outgassing to remove adsorbed

moisture. This specimen insures that neither the sample mount nor the gold film themselves are responsible for the observed electron emission.

## Discussion

A simple model for CVD low field cold electron emission emerges from these observations, which suggests that a minimum field is necessary to achieve dielectric breakdown. Figures 3 and 4 show each stage of the experiment, with a value for the applied field suggested by our simple model. In figure 3a the [100] diamond surface is shown in profile in front of the microscope optics, which is at a high positive voltage  $V$  a distance  $D$  ( $\sim 4\text{mm}$ ) away. Before the gold film is deposited the applied field is  $E=V/D$ . When the gold film is deposited (figure 3b), the layer formed is smooth, but not continuous on a macroscopic scale. (If the film were continuous, no field emission threshold would occur, and a PEEM image of metallic gold would be observed under UV illumination.) The gold layer causes an enhancement leading to a field at the film of  $E'=V/d$ .

The model for this enhancement is illustrated in figure 4. The insulating diamond film acts as a dielectric, as in a capacitor, and graphitic inclusions act as conducting paths [8,11]. The gold layer charges up to the lens potential  $V$  (figure 4a). The field at the film is now  $E'=V/d$  ( $d$  = the film thickness  $\sim 100\mu\text{m}$ ), a field enhancement of a factor of  $\sim 100$  in our experiments. The "turn-on" field of  $30\text{kv/cm}$  indicates that a minimum critical voltage must be reached in order to cause breakdown. When the voltage is reached (figure 4b), the diamond film breaks down under the "high" field, shorting the "capacitor", and hot electron emission, collected by the microscope as an image, results. After heating the film, the gold is still present, but the ball-shaped geometry does not provide the field enhancement needed for dielectric breakdown

(figure 4c). Since the process is completely reversible, no chemical alteration of the film by the gold layer takes place.

Clearly, it is necessary that the film become conductive to allow electron emission; this is possible if there is conduction due to dielectric breakdown [18,19]. We suggest that the minimum applied field necessary to induce dielectric breakdown is related to the impurity content of the film. In doped films, or in "low-quality" films (those with low resistivity/high number of graphitic inclusions) such as the MWPCVD faceted (111) sample, conduction paths already exist in the form of graphite, and low fields are enough to cause breakdown across the diamond and subsequent emission. In non-emitting films such as the textured [100] sample, high fields must be reached to cause dielectric breakdown and emission to occur. Recent measurements [18] of dielectric breakdown in [100] textured CVD diamond films show that the breakdown occurs at about  $1 \times 10^6$  V/cm. The field enhancement that is attribute to the gold coating combined with the breakdown threshold observed in our emission microscope on the MWPCVD [100] film is also about  $10^6$  V/cm.

In high resistivity films, the fact that the "gold" activation process is reversible indicates that the conductive paths created in our [100] textured diamond experiment do not cause permanent damage to the film structure or chemistry. In other words, new breakdown pathways that subsequently enabled conduction at lower fields were not created.

Field emitters based on a diode structure have been reported for diamond [3] and other materials [21-23]. Metallic conduction is believed to take place through population of the conduction band of the insulating material of these emitters. Other views have been developed by Latham and co-workers, based on non-metallic conduction in "hot electron" emitters [10,11,24]. In ex situ measurements, both in our lab [25] and others [11,12], Fowler-Nordheim-like behavior is observed for CVD diamond electron emission. Further investigations into the detailed emission mechanism will probably depend on deviations from this

behavior, possibly as a function of temperature or dopant level.

### Summary

The reversible "activation" of previously non-emitting CVD diamond films presented here is consistent with field emission by non-diamond conduction paths that are created during dielectric breakdown of the diamond film and possibly assisted by graphite conductors in the film. A minimum applied field for emission is necessary. A simple, qualitative model of the effect of the gold layer suggests that the gold film acts as an electrode for direct application of the accelerating field for electron emission to the front surface of the film rather than over an acceleration gap of several mm, providing a field enhancement large enough to cause dielectric breakdown in highly resistive diamond films.

The gold coating should quench effects due to surface crystallographic orientation, surface electronic states and surface termination by specific adsorbates, thus negative electron affinity effects probably do not play a major role in low field cold electron emission from CVD diamond.

### Acknowledgements

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## FIGURE CAPTIONS:

Figure 1: A typical field emission image of the gold coated textured diamond [100] film. Bar is 40  $\mu\text{m}$ .

Figure 2: SEM micrographs of the gold coated textured diamond [100] film. a) Before heating. Film is uniform and smooth. b) After heating. Film appears discontinuous and rough. c) Close-up of [100] face. Gold forms individual sub-micron sized spheroids.

Figure 3: The diamond [100] surface shown in profile in front of the microscope optics. a) The uncoated sample. The sample experiences a field  $E=V/D$  where  $D$ , the distance from the lens to the film, is 4mm, and  $V$ , the microscope potential, ranges from 0-20kV. No emission occurred for this configuration. b) After gold coating field emission is observed. The gold islands cause the film to experience a field  $E=V/d$  where  $d$ , the thickness of the film, is 100 $\mu\text{m}$ , an enhancement of a factor of  $\sim 100$ .

Figure 4: The gold forms smooth islands, discontinuous on a macroscopic scale. These isolated conducting islands enhance the field at the surface of the diamond. a) The network of conducting graphitic inclusions and insulating diamond acts as a capacitive circuit. For voltages less than that required for dielectric breakdown, there is no emission. b) When breakdown occurs, conduction paths form, and electrons are emitted. c) The balled-up gold does not provide field enhancement, and field emission stops.

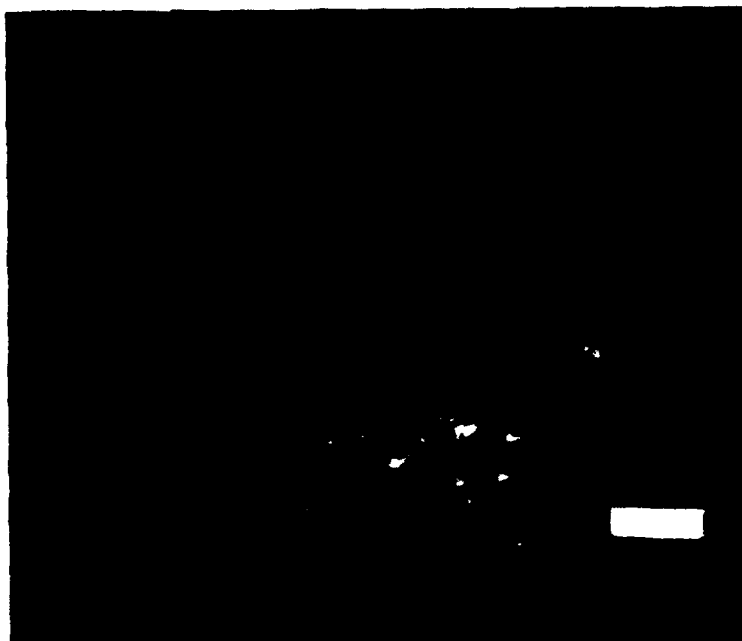


Fig 1

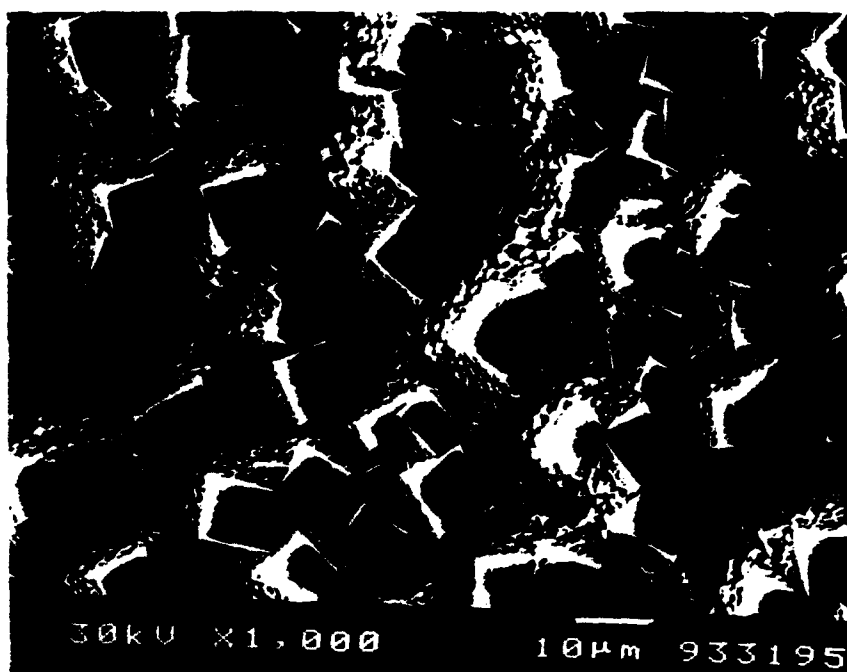


Fig 2a

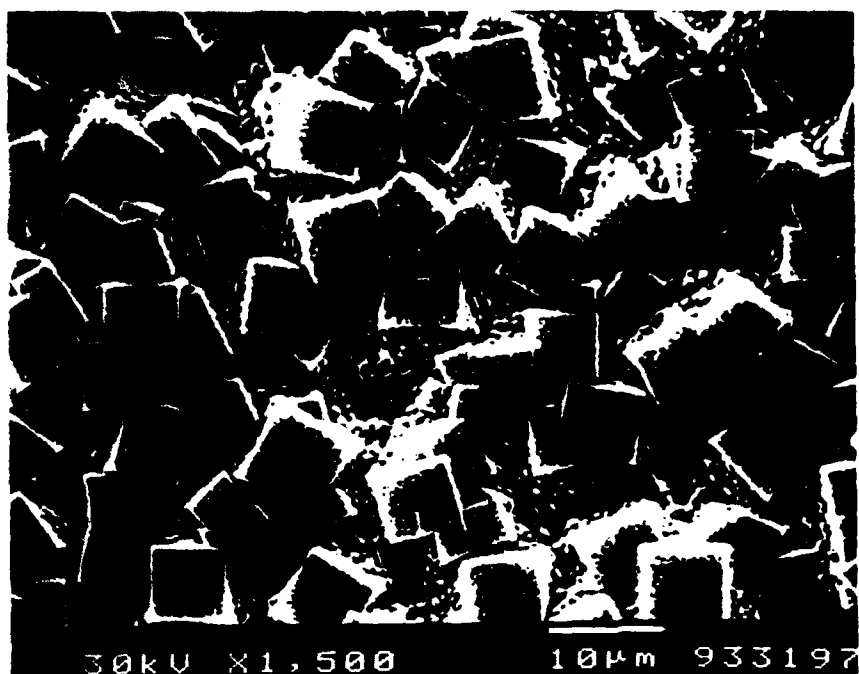


Fig 2b

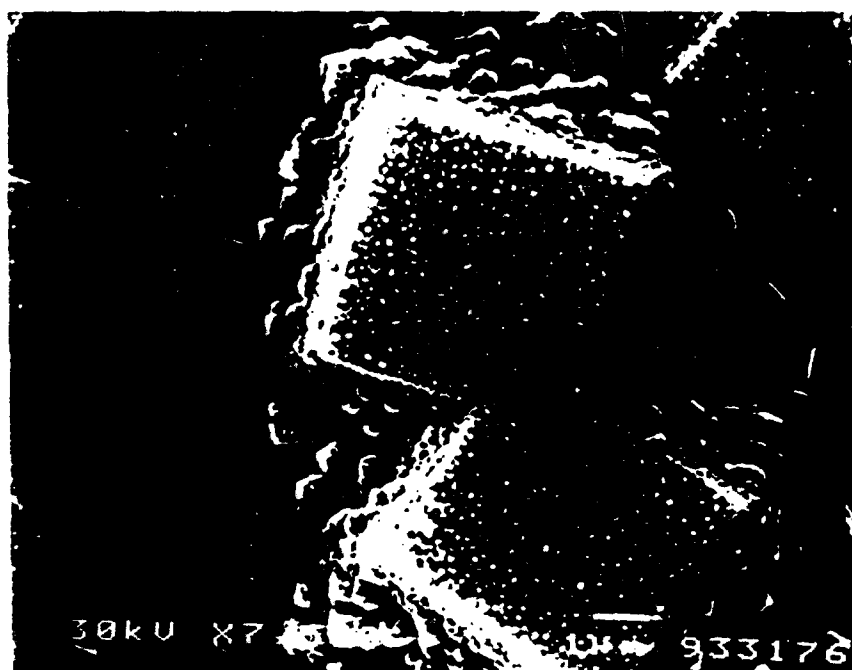


Fig 2c

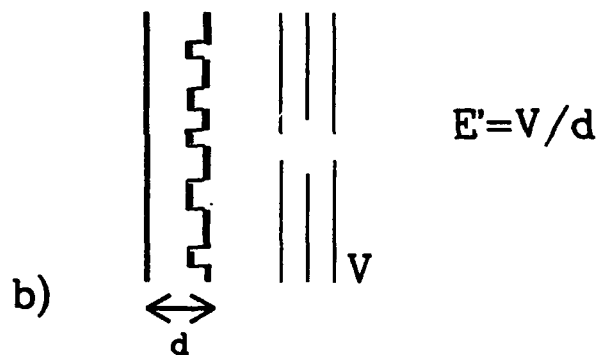
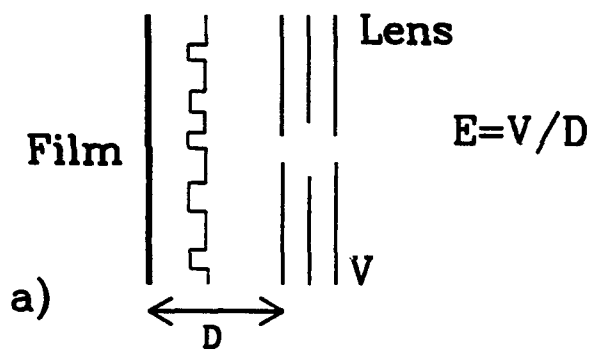


Fig 3

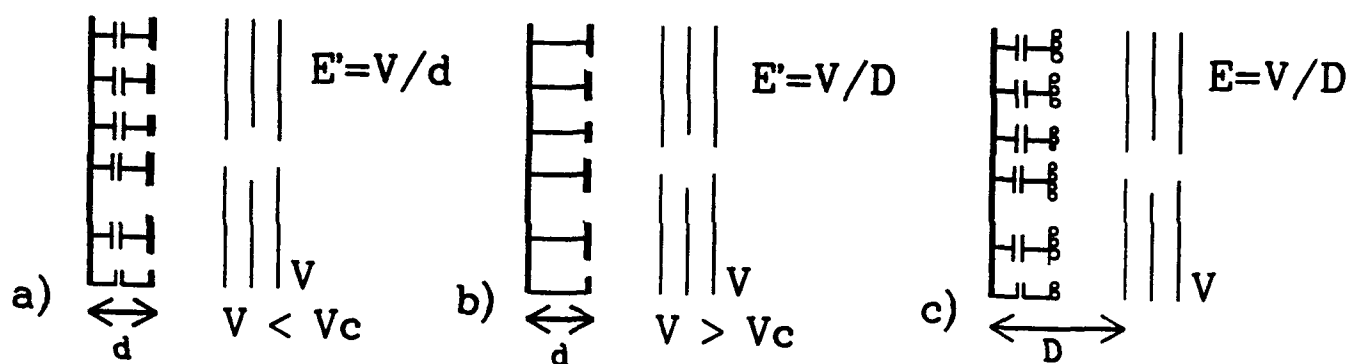


Fig 4